Laboratory Manual
Chemistry (UCB009)

for

B.E. /B.Tech-1st Year

THAPAR INSTITUTE
OF ENGINEERING & TECHNOLOGY
(Deemed to be University)

Department of Chemistry & Biochemistry

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Preface

A Brief Overview of the Chemistry (UCB009) Course & Online Laboratory Manual

Course Objective: The course aims at elucidating principles of chemistry in industrial systems, water treatment, engineering materials and analytical techniques.

Chemistry (UCB009) is a **4.0 credits** course which comprises lectures, tutorials and practicals. Typically, there are 3 lectures (of 1 hour each) and 1 practical (2 hours) per week (i.e., L T P **Cr** = 3 0 2 **4.0**). Attending all of the classes is mandatory and the attendance should be maintained at ≥75% (combined number of hours of L and P).

The "Practicals" are conducted in the Chemistry laboratories (designated as 'CBOL' CBCL and 'CBTL'; located on the ground and first floor of 'G'-block) at the School of Chemistry and Biochemistry (SCBC), TIET, which gives students an opportunity to carry out experiments related to important chemical concepts that they have learned during the course. During practicals, the students are exposed to various instruments, experimental techniques, and laboratory safety practices. Students are expected to perform 10 experiments and are evaluated for practical examination just before the start of end semester examination.

The **Online Lab Manual** serves as an important source of information for the Chemistry Practicals. The manual consists of detailed information on writing and maintaining a good laboratory notebook, experimental procedures, theories, chemical structures, and safety precautions required for each experiment. The students are required to go through each experiment and its related information.

The outcome of practical's and the list of the experiments to be performed

Laboratory Work Outcome: Students will perform experiments involving the use of pH meter, conductivity meter, potentiometer, and colorimeter. They will also learn to determine the hardness, alkalinity, chloride, and iron content in aqueous medium. The 'Practical' component of this course consists of 10 experiments that can be broadly classified into two types:

I. Volumetric Analysis-based
II. Equipment/Instrument-based

Following is a list of experiments in both categories:

I. Volumetric Analysis-based Experiments

1. To find out total alkalinity and chloride content in the given water sample.
2. To find the temporary and permanent hardness of water sample by complexometric titration using standard EDTA solution.
3. To determine the amount of Fe^{+2} and Fe^{+3} ions by permanganatometry.
4. To determine the amount of NaOH and Na_2CO_3 present in the same solution.
5. To determine the molecular weight of a polymer (polystyrene) by using Ostwald's viscometer.
6. To determine the copper content of a given sample of copper ore solution using 0.1 N sodium thiosulphate iodometrically.

II. Equipment/Instrument-based Experiments

1. To determine the strength of given sodium hydroxide solution by titration with standard hydrochloric acid conductometrically.
2. Determine pK_a value of acetic acid by pH-metric titration.
3. To titrate potentiometrically FAS solution against potassium permanganate and to determine the standard electrode potential of Fe^{2+} / Fe^{3+} system.
4. Spectrophotometric determination of Fe^{2+} with 1,10-phenanthroline.
5. Determination of cloud and pour point of a given sample.

Maintaining a Laboratory Notebook: Tips on Writing, Preparing Graphs etc.

Keeping a laboratory notebook, which serves as a permanent record, is an extremely important part of Science and Engineering curriculum and gives detailed information about the objective, procedure, observations, calculations and result of an experiment that is carried out. Please ensure that the **writing is complete, thorough, and legible**.

❖ Organization of a good lab notebook

Conventionally, a chemistry laboratory notebook comprises two kinds of pages namely, a 'blank page' and a 'ruled page' that are adjacent to each other wherein experimental details are filled. In addition to these, a title page and an index page are also present at the beginning of the notebook. Following are the universally acknowledged rules of writing and maintaining a good, comprehensive laboratory notebook:

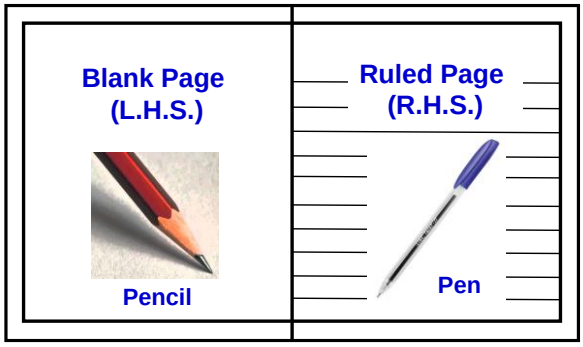


Fig. 1: Schematic representation of the pages in a conventional laboratory

1. On the **title** page, please write your **name, roll number, group code, course code, and the course name**.

2. The **index page** essentially serves as a **'table of contents'** and gives a very brief overview of the sequence of list of experiments that are performed on a particular date. ***The students are advised to fill up the index page promptly whenever they carry out an experiment***.

3. The student **MUST** fill up the **date, experiment number** and the **page number** on the top of the ruled page. A chronological, page-wise list of experimental write-up is being tabulated:

Blank Page (L.H.S.)	Ruled Page (R.H.S.)
Date:_____ (date on which the experiment is actually performed)	
Experiment No. _____	
Experiment : (Name of the Experiment)	**Experiment /Aim:** (Name of Experiment)
Apparatus:	**Chemical equations:** (where applicable)
Chemicals required:	**Procedure:**
Indicator: (Where applicable) **End point: (i.e., Change in colour at the end point)**	**Precautions:**
Observations: (with proper units)	**Application in daily life:**
Calculations: (Based upon the values obtained during experimentation/observation)**/Graph**	
Result: (with proper units; Report the result(s) as required in the aim of experiment)	

❖ Drawing a graph properly and interpreting results

As mentioned earlier, the student is required to perform a few **instrument-based experiments**. After the data is noted/ recorded, the students will be required to **plot the data-points** on a graph sheet using a pencil and **join the points** which will result in a particular **graphical pattern**. The graph, hence generated, will be consequently used to **interpret the results**.

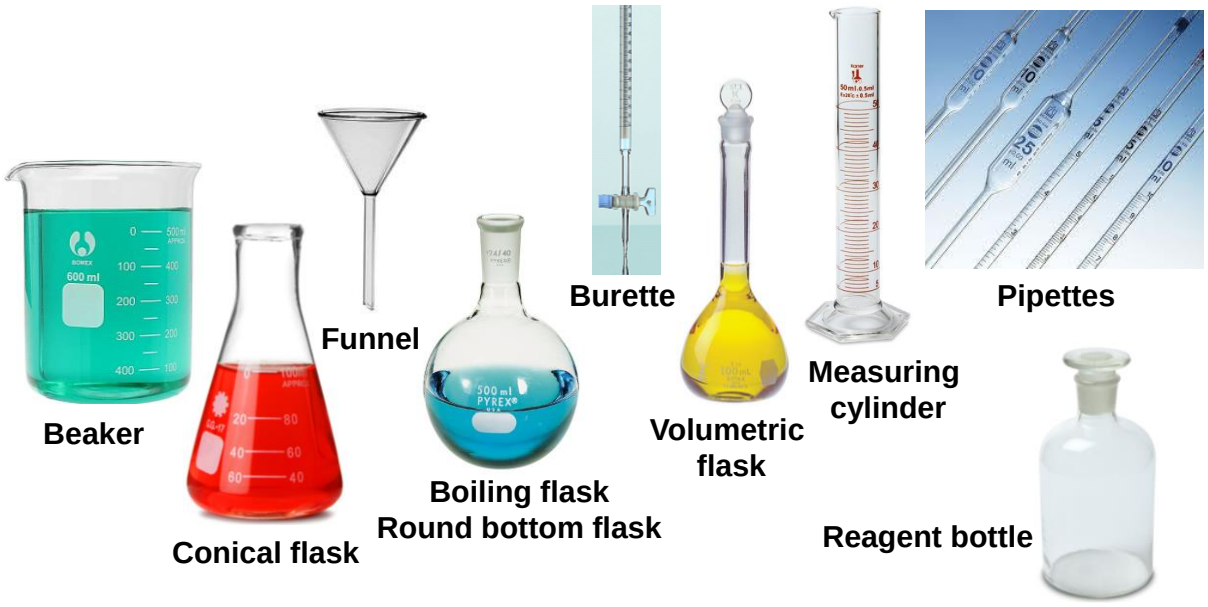
A few tips for the preparation of a good graph:

1. While labeling the axes (X, Y), please ensure that you have included the units of the variables properly, wherever it is applicable. A graph without any units is obscure and does not give an idea about the measureable quantities, volumes etc. that are required and used in an experiment.
2. Mark the XY-data-points on the graph.
3. Interpretation of an accurate and precise result from a graph is very important for an instrument-based experiment. However, while joining the data-points, please ensure from your instructor and/or teaching associate whether a ruler/scale is needed by you. Here are a few basic rules for joining the data-points:

- Certain graphical analysis requires drawing more than one slope and finding their intersection points.
- **REMEMBER**: When you join the data-points using a ruler/scale to draw a straight line or a slope, please **DO** ensure that the line/slope passes through a maximum number of data-points. It does not matter and it is absolutely **NOT** essential that all of the data-points have to fall on the straight line. It is **OKAY** if two/three data-points fall outside the line/slope. So, place your ruler in a few different ways to find out the best possible way of drawing a line/slope.
- A few graphs require joining the data-points by freehand drawing. Please **DO NOT** worry if one of the data-points does not fall onto the graph. It could be an experimental error/artefact which is caused involuntarily.

Commonly used Glassware in this Practical Course

Glassware are containers made up of glass and are commonly used in a chemistry laboratory because of the following reasons: Glass is (a) chemically resistant (b) heat-resistant and (c) transparent. While carrying out the experiments, you will frequently use some glassware in the laboratory. It is always important and useful to know their proper names. Also, **please make sure that you wash/clean any glassware thoroughly before, and after each experiment.**



General precautions / important points to take care during experimentation

- Rinse the pipette (with the solution to be transferred to titration flask).
- Rinse the burette (with the solution to be taken/filled in the burette).
- Donot rinse the conical/titration flask.
- Upper meniscus to be read for coloured solutions.
- Lower meniscus to be read for colourless solutions.
- End point (signifies the change in colour of the solution e.g., from pink to colourless).

Chemical Structures of Commonly Used Indicators

Indicators are defined as chemical sensors/detectors, required in extremely small quantities in a solution, which detect the changes in pH, redox potential, metal-ion complex formation, etc. Here, we shall discuss primarily two types of indicators that are going to be used in the practical's:

(1) pH-based indicators: These indicators are weak, conjugated organic acids that detect the changes in pH of a solution, especially after the completion of a titration. Since the indicator is a weak acid (HIn), an equilibrium will be established as this acid dissociates in an aqueous solution and the dissociation constant can be represented as shown in the schematic representation (Fig. 2). According to the Le Chatelier's principle, at lower pH (acidic condition),

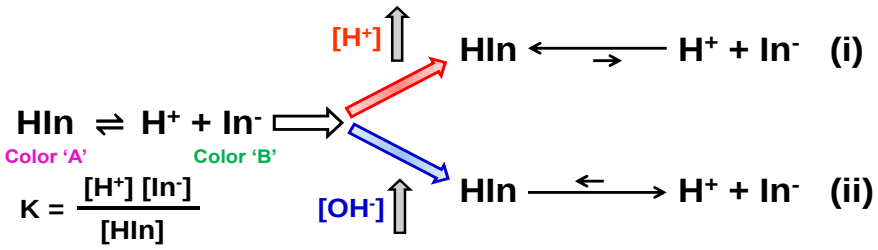
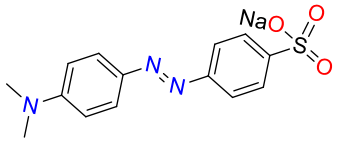
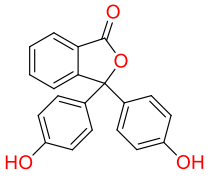


Fig. 2: Schematic representation of the effects of pH on a pH-based indicator. 'K' is the dissociation

the species 'HIn' will be predominant (eq. (i)) in the aqueous solution which can be detected by its color 'A' (Fig. 2). On the other hand, at higher pH (alkaline condition), the species 'In^-' will be predominant (eq. (ii)), since the H^+ ions (released by HIn) will combine with OH^- to form water and hence, will get depleted progressively resulting in further enhancement in the dissociation (forward reaction). The predominance of In^- can be easily detected by its color 'B' (Fig. 2) in the aqueous solution. During an acid-base titration, a pH-based indicator is used that changes its color exactly at the end-point of the titration. The changes in color occur due to changes in the extent of conjugation upon protonation-deprotonation of one or more of the chemical moieties/substituents in HIn. However, this must be noted that there **DOES NOT** exist any universal pH indicator which works across the entire pH range.

Following is a list of a few pH-based indicators:

S. No.	Names	Chemical structure of pH indicator	pH range	Color (pH) Acidic to Basic
1.	Methyl Orange		3.1 – 4.4	Red to Yellow
2.	Phenolphthalein		8.2 - 10	Colorless to Pink

(2) Complexometric indicators: These indicators are weak, conjugated organic acids that detect binding of a metal-ion to a ligand leading to a metal-ligand complex formation in a solution. Typically, these indicators are functional at a given pH of the solution and they undergo a color change as the binding occurs. For example, Eriochrome Black-T (EBT) detects the complex formation between Ca^{2+}/Mg^{2+} and Ethylenediaminetetraacetic acid (EDTA) at pH 10 and its color changes from wine-red to blue. The utility and properties of EBT (in detail) is given in experiment no. 2 of this manual.

List of Experiments for B.E./B.Tech (First year)

1. To find out total alkalinity and chloride content in a given water sample.
2. To find the temporary and permanent hardness of the water sample by complexometric titration using standard EDTA solution.
3. To determine the amount of Fe^{+2} and Fe^{+3} ions by permanganatometry.
4. To determine the amount of NaOH and Na_2CO_3 present in the same solution.
5. To determine the molecular weight of a polymer (polystyrene) by using Ostwald's viscometer.
6. To determine the copper content of a given sample of copper ore solution using 0.1 N sodium thiosulphate iodometrically.
7. To determine the strength of given sodium hydroxide solution by titration with standard hydrochloric acid conductometrically.
8. Determine pK_a value of acetic acid by pH-metric titration.
9. To titrate potentiometrically FAS solution against potassium permanganate and to determine the standard electrode potential of Fe^{2+} / Fe^{3+} system.
10. Spectrophotometric determination of Fe^{2+} with 1,10-phenanthroline.
11. Determination of cloud and pour point of a given sample.

EXPERIMENT NO. 1

EXPERIMENT: Find out the total alkalinity and chloride content in a given water sample.

APPARATUS: Pipette, burette, beakers, conical flask, funnel, burette stand and clamp.

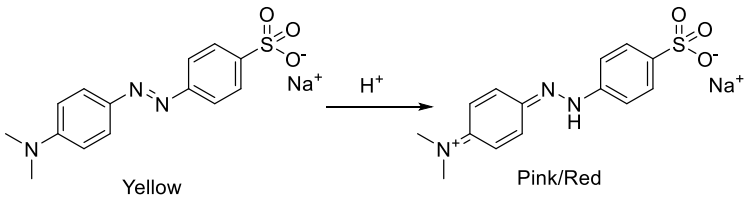
CHEMICALS: Water samples, Potassium Chromate (K_2CrO_4) indicator, Silver Nitrate ($AgNO_3$), methyl orange and sulphuric acid (H_2SO_4).

THEORY: Alkalinity of water is due to the presence of hydroxides, carbonates and bicarbonates of the salts of calcium, magnesium, sodium and potassium. Similarly, the chloride content of water is due to the presence of chloride ions of these cations. Total alkalinity is estimated by titrating a known volume of water against a standard acid (N/20 H_2SO_4) using methyl orange as indicator in the neutral medium.

$$CO_3^{2-} + 2H^+ \longrightarrow CO_2 + H_2O$$

$$HCO_3^- + H^+ \longrightarrow CO_2 + H_2O$$

$$OH^- + H^+ \longrightarrow H_2O$$



choride content is estimated by titrating a known volume against a standard silver nitrate solution (N/100) using Potassium Chromate (K_2CrO_4) as indicator.

PROCEDURE

(i) Determination of total alkalinity of tap water

1. Wash, rinse, and fill the burette with N/20 H_2SO_4.
2. Transfer 25 ml of the given water sample to the titration flask. Add 2-3 drops of methyl orange and titrate it against N/20 H_2SO_4 till the color changes from ***yellow*** to ***light pink***, as an end point.
3. Note the volume of the solution used and repeat the titration at least 5 times and take the mean of the closely related readings (x ml).

(ii) Determination of choride contents of water sample

1. Take 10 ml of water sample in a titration flask.

2. Add 3-4 drops of Potassium Chromate (K_2CrO_4) and titrate against N/100 Silver nitrate from the burette till the color changes from **yellow to brick red**.

3. Note the volume of the solution used and repeat the titration at least 5 times and take the mean of the closely related readings (y ml).

OBSERVATIONS

Let the volume of N/20 H_2SO_4 used = x ml

Let the volume of N/100 $AgNO_3$ used = y ml

GENERAL CALCULATIONS

(i) Alkalinity

Applying the normality equation

(Tap water) (H_2SO_4)

$N_1V_1 = N_2V_2$

100 ml of tap water (of normality N_1) = x ml of N/20 H_2SO_4

$$N_1 = \frac{N_2V_2}{V_1} = \frac{0.05\,x}{25} = \frac{x}{500}$$

Eq. wt. of $CaCO_3$ = 50 ; Amount of $CaCO_3$ (gm/L) = Normality × Eq. wt.

$$= \frac{x}{500} \text{x } 50 = \frac{x}{10}$$

Amount of $CaCO_3$ (mg/1000 mL) $= \frac{x}{10} \text{x } 1000\ ppm$

(ii) Chloride Content

Applying the normality equation,

(Tap water) ($AgNO_3$)

$N_1V_1 = N_2V_2$

10 ml of tap water (of normality N_1) = y ml of N/100 $AgNO_3$ solution

$$N_1 = \frac{N_2V_2}{V_1} = \frac{y}{1000}$$

Eq. wt. of Cl^- = 35.5

Chloride Content (gm/L) = Normality × Eq. wt.

$$= \frac{y}{1000} \text{ x } 35.46$$

Chloride Content (mg/1000 mL) = $y \times 35.46$ *ppm*

RESULTS

Amount of total alkalinity in water sample _____ ppm of $CaCO_3$

Amount of chloride content in water sample _____ ppm

Expected CLOs/Daily life application: Alkalinity and chloride ion is important for aquatic life as it helps in maintaining pH. However, highly alkaline water can corrode the pipelines. Alkaline water is helpful in recovering petroleum too. Also, to maintain quality of potable water, quantification of both alkalinity and chloride ions is crucial.

EXPERIMENT NO. 2

EXPERIMENT: To find the temporary and permanent hardness of water sample by complexometric titration using standard EDTA solution.

APPARATUS: Pipette, burette, beakers, conical flask, funnel, burette stand and clamp.

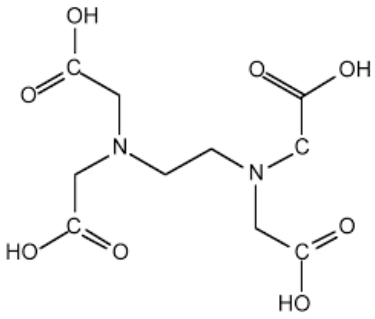
CHEMICALS: Water samples, ethylenediaminetetraacetic acid (EDTA), Eriochrome Black-T (EBT) indicator, ammonium hydroxide-ammonium chloride buffer of pH 10

THEORY: Hardness of water is due to the presence of soluble salts of Ca and Mg. It is an important parameter to judge the quality of water. Determination of hardness of water by EDTA titration is a very accurate method based on the fact that when Eriochrome black-T (blue dye) is added to the hard water (at about pH 10), it gives a wine red colored unstable complex with Ca^{2+}/Mg^{2+} ions.

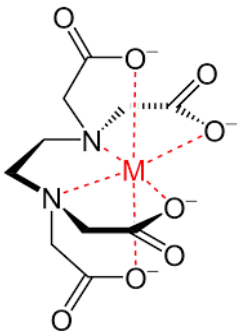
Ca^{2+}/Mg^{2+} + Eriochrome black-T **(blue dye)** ⟶ Ca^{2+}/Mg^{2+} Eriochrome black-T complex **(unstable wine red complex)**

Temporary hardnesss in a water sample is caused by bicarbonates of hardness producing ions (Ca^{2+} and Mg^{2+}). This can be removed by prolonged boiling due to decomposition of bicarbonates with the evaluation of CO_2 and simultaneous precipitation of the respective carbonates. When EDTA (ethylene diammine tetraacetic acid) solution is added to the hard water (with permanent or temporary hardness), the unstable wine red complex of Ca^{2+}/Mg^{2+}-Eriochrome black-T breaks and a stable complex of Ca^{2+}/Mg^{2+} with EDTA is formed resulting in change of color of the solution from wine red to blue at the end point.

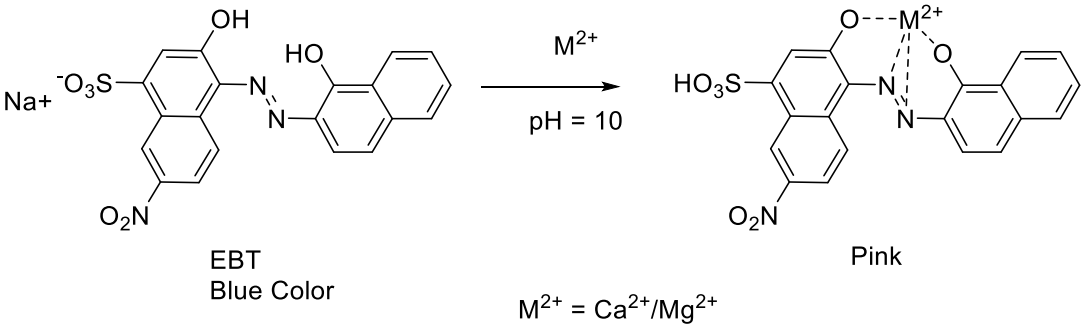
Ca^{2+}/Mg^{2+} Eriochrome black-T complex **(unstable wine red complex)** + EDTA ⟶ Ca^{2+}/Mg^{2+} EDTA complex **(colorless)** + Eriochrome black-T **(original blue dye)**



EDTA



EDTA-Metal complex



PROCEDURE

Preparation of standard hard water: Dissolve 1 gm of pure dry $CaCO_3$ in minimum quantity of dilute HCl. Evaporate the solution to dryness on a water bath to remove excess of acid. Dilute the contents with distilled water to make 1L. Each mL of this solution contains 1 mg of $CaCO_3$, i.e., hardness of this solution is 1000 ppm (0.01 M). This solution is used to standardize the EDTA solution.

Standardization of EDTA

1. Rinse the titration flask with distilled water and transfer 10mL of the standard hard water sample (0.01 M) into it using a pipette.
2. Add about 2-3mL of ammonia/ammonium chloride buffer solution and a very small amount (2-3 drops) of the EBT indicator. The color of solution becomes wine red.
3. Titrate the solution against the EDTA solution, till the wine ***red color changes to blue.*** Note the burette reading (V_0 mL).
4. Repeat the procedure until three concordant readings are obtained.

Determination of total hardness of water sample

1. Rinse the titration flask with distilled water and transfer 10mL of the water sample into it using a pipette.

2. Add about 2-3mL of ammonia/ammonium chloride buffer solution and a very small amount (2-3 drops) of the EBT indicator. The color of solution becomes wine red.

3. Titrate the solution against the standard EDTA (Molarity, M_1) solution, till the wine ***red color changes to blue***.

4. Note the volume of the solution required to complete the titration. Let this volume used be V_1. This corresponds to the total hardness.

Determination of permanent hardness

1. Rinse the titration flask with distilled water and pipette 10mL of the boiled sample into it.

2. Add about 2-3mL of ammonia/ammonium chloride buffer solution and a very small amount (2-3 drops) of the EBT indicator. The color of solution becomes wine red.

3. Titrate with standard EDTA solution till the color changed from ***wine red to blue*** at the end point.

4. The titre value corresponds to permanent hardness (V_2).

Determination of temporary hardness

Difference between the two values ($V_1 – V_2$) corresponds to temporary hardness

OBSERVATIONS

(i) Standardization of EDTA solution

Volume of 0.01 M standard hard water solution taken for each titration = 10 mL

Sr. No.	Burette reading (mL)		Volume of EDTA used (mL)
	Initial	Final	
1.			
2.			
3			
4			

Mean volume of EDTA used (V_0)= _____ (mL)

(ii) Determination of total hardness

Volume of hard water sample (unknown) taken for each titration = 10 mL

Sr. No.	Burette reading (mL)		Volume of EDTA used (mL)
	Initial	Final	
1.			
2.			
3			
4			

Mean volume of EDTA used (V_1)= _____ (mL)

(iii) Determination of permanent hardness

Volume of boiled hard water sample taken for each titration = 10 mL

Sr. No.	Burette reading (mL)		Volume of EDTA used (mL)
	Initial	Final	
1.			
2.			
3			
4			

Mean volume of EDTA used (V_2)= _____ (mL)

GENERAL CALCULATIONS

(i) Determining the molarity of EDTA solution

Applying the molarity equation

(Standard Hard water) (EDTA)

$0.01 \times 10 = M_1 \times V_0$

Molarity of the EDTA, $M_1 = (0.01 \times 10)/V_0$

(ii) Determination of total hardness

Molarity of EDTA = M_1

Applying the molarity equation

(Hard water) (EDTA)

$M_2 \times 10 = M_1 \times V_1$

Molarity of the hard water, $M_2 = (M_1V_1)/10$

Hardness of water sample, Y = Molarity × Molecular weight of $CaCO_3$

Hardness of water sample, Y = $(M_1V_1)/10 \times 100$ (Molecular weight of $CaCO_3$) gm/L

= $(M_1V_1)/10 \times 100$ (Molecular weight of $CaCO_3$) × 1000 mg/L

Total Hardness = Y ppm (mg/L)

(iii) <u>Determination of permanent hardness</u>

Again apply the molarity equation

(Boiled Hard water) (EDTA)

$M_3 \times V_1 = M_1 \times V_2$

M_3 = (Molarity of hard water due to permanent hardness)

Molarity of the hard water, $M_3 = (M_1V_2)/10$

Permanent hardness of water sample, Z = Molarity × Mol. wt. of $CaCO_3$

Permanent hardness of water sample, Z = $(M_1V_2)/10 \times 100$ (Mol. wt of $CaCO_3$) gm/L

= $(M_1V_2)/10 \times 100$ (Mol. wt of $CaCO_3$) × 1000 mg/L

Permanent hardness = Z ppm (mg/L)

(iii) <u>Temporary hardness</u>: Total hardness – Permanent hardness = (Y − Z) ppm

<u>RESULTS</u>: Total hardness = Y ppm

Permanent hardness = Z ppm

Temporary hardness = (Y − Z) ppm

<u>PRECATIONS</u>

(1) Wash the titration flask with distilled water each time, before transferring hard/ sample water solution.

(2) Continue the titration till the complete removal of wine-red tinge in the solution.

Expected CLOs/Daily life application: Determination of hardness of water can help in industrial settings, where water hardness is monitored to avoid costly breakdowns in boilers, cooling towers, and other equipment. High calcium levels also cause irritation in eyes. Hence, quantification of ions in potable water is necessary.

EXPERIMENT NO. 3

EXPERIMENT: To determine the amount of Fe^{+2} and Fe^{+3} ions by permanganatometry.

APPARATUS: Pipette, burette, beakers, conical flask, funnel, burette stand and clamp.

CHEMICALS: Mohr's salt solution (Ferrous ammonium sulfate; $FeSO_4(NH_4)_2SO_4.6H_2O$)), permanganate ($KMnO_4$) and sulphuric acid (H_2SO_4).

THEORY: Mn^{7+} oxidises Fe^{+2} in acidic medium to Fe^{3+} and itself gets reduced to divalent chromium (Mn^{2+})

$$MnO_4^- + 8H^+ + 5Fe^{2+} \rightleftharpoons Mn^{2+} + 5Fe^{3+} + 4H_2O$$

$KMnO_4$ acts as a self-indicator. If Fe^{3+} is present in the original solution, it can be reduced by boiling the solution with zinc pieces in acidic medium and can be titrated with standard $KMnO_4$. The end point in this case corresponds to presence of both Fe^{+2} and Fe^{+3} ions in the solution.

PROCEDURE

(i) Standardization of $KMnO_4$

1. Transfer 10 mL of the standard 0.1 N ferrous ammonium sulfate (FAS) solution to a clean conical flask using a pipette.
2. Add 5 mL of 4 N sulphuric acid.
3. Titrate the solution against $KMnO_4$ solution taken in a burette. The color of the solution changes from ***colorless to pink***.
4. Note the volume of the solution used and repeat the titration at least 5 times and take the mean of the closely related readings (V_1).

(ii) Determination of Fe^{+2}

1. Pipette out 10 ml of the given solution in the titration/ conical flask.
2. Add 5 mL of 4 N sulphuric acid.
3. Titrate the solution against $KMnO_4$ solution taken in a burette. The color of the solution changes from ***colorless to pink***.

4. Note the volume of the solution used and repeat the titration at least 5 times and take the mean of the closely related readings (V_2).

(iii) Determination of Fe^{+2} and Fe^{+3} (Total iron content)

1. Pipette out 10 ml of aqueous solution into the conical flask. [The given solution has already been boiled with 2-3 grams of zinc pieces and 5 mL of dilute H_2SO_4 to reduce the Fe^{3+} to Fe^{2+}].
2. Add 5 ml of 4N H_2SO_4.
3. Titrate it with standard $KMnO_4$ solution till the solution turns from ***colorless to pink***.
4. Note the volume of the solution used and repeat the titration at least 5 times and take the mean of the closely related readings (V_3).

GENERAL CALCULATIONS

GENERAL CALCULATIONS

(i) Normality of $KMnO_4$ solution

Apply the Normality equation

($KMnO_4$) (FAS)

$N_1V_1 = N_2V = 0.1\ N \times 10\ mL$

Normality of the $KMnO_4$ $N_1 = (0.1 \times 10)/V_1 = S\ (N)$

(i) Determination of Fe^{+2}

Vol. of solution taken = 10 mL

Vol. of $KMnO_4$ solution used = V_2 mL

Normality of $KMnO_4$ = S N

Normality of Fe^{2+}

$$N_1 = \frac{V_2 \times S}{10}$$

Strength of Fe^{2+} = N_1 x Eq. wt.

= N_1 x 56 gm/L

(ii) Determination Fe^{+3} in a mixture of Fe^{+2} and Fe^{+3}

Vol. of solution taken = 10 mL

Vol. of $KMnO_4$ solution used = V_3 mL

Normality of $KMnO_4$ = S N

Normality of total Fe

$$N_1 = \frac{V_3 \text{ x } S}{10}$$

Strength of total Fe = N_1 x Eq. wt.

= N_1 x 56 gm/L

Strength of Fe^{+3} ions = Normality × Eq. wt.

$$= \frac{(V_3 - V_2) \text{ x } S \text{ x } 56}{10}$$

RESULTS: The amount of Fe^{+2} = _____ gm/L ; and the amount of Fe^{+3} = _____ gm/L.

Expected CLOs/Daily life application: In qualitative and quantitative determination of Fe^{2+} and/or Fe^{3+} present in an ore or compound, water sample etc.

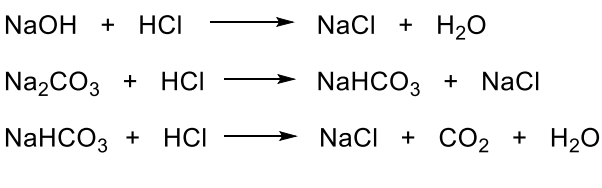
EXPERIMENT NO. 4

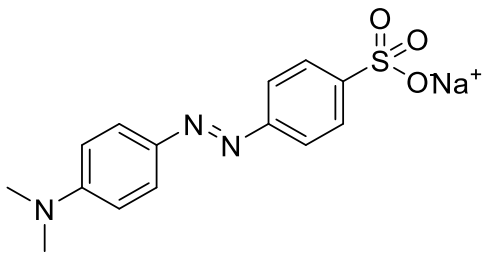
EXPERIMENT: To determine the amount of NaOH and Na_2CO_3 present in the same solution.

APPARATUS: Pipette, burette, beakers, conical flask, funnel, burette stand and clamp.

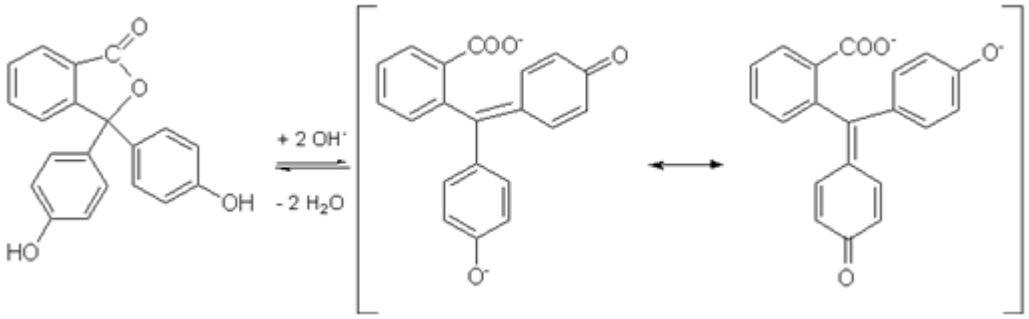
CHEMICALS: Sodium carbonate (Na_2CO_3), sodium hydroxide (NaOH), hydrochloric acid (HCl), methyl orange and phenolphthalein.

THEORY: When a mixture of NaOH and Na_2CO_3 is titrated against a standard HCl solution, colour of solution changes from yellow to pink (using methyl orange as an indicator) due to complete neutralization of both the alkalis at pH ≈ 4. However, when the mixture is titrated using phenolphthalein as an indicator, the colour of the solution changes from pink to colorless due to complete neutralization of NaOH and half neutralization of Na_2CO_3 (i.e., upto the conversion of Na_2CO_3 to $NaHCO_3$) at pH ≈ 8. The difference of two titre values gives the amount of HCl required for half neutralization of Na_2CO_3 while the difference of first titre value and twice the second titre value gives the amount of HCl required for NaOH neutralization.





Methyl Orange



Colorless **Pink**

Phenolphthalein

PROCEDURE

(i) Standardization of HCl

1. Transfer 10 mL of standard 0.1N Na_2CO_3 solution in a clean conical flask using a pipette.
2. Add 2 drops of methyl orange indicator. Titrate the solution against HCl from the burette.
3. The color of the solution changes from ***yellow to pink*** **(end point).**
4. Note the volume of the solution used and repeat the titration at least 5 times and take the mean of the closely related readings (V_o).

(ii) Determination of NaOH and Na_2CO_3 content

1. Transfer 10 mL of mixture of alkali solution into a conical flask.
2. Add 2-3 drops of phenolphthalein indicator. The solution ***becomes pink*** in color.
3. Titrate the solution with standard HCl from burette while the solution becomes ***colorless***.
4. Note the titre value and this is the phenolphthalein end point [P]. **To the same solution,** add 2-3 drops of methyl orange indicator and continue the titration with HCl, until a sharp color change occurs from ***yellow to red*** at the end point.
5. This titre value i.e., the total volume of HCl run down from the beginning of the experiment to the methyl orange and point is noted and this is the methyl orange end point [M].

OBSERVATIONS

Volume of the Na_2CO_3 /mixture (of NaOH and Na_2CO_3 solution) taken in titration flask = 10 ml

Volume of HCl used for:

1. Standardization of HCl = V_o
2. Phenolphthalein end point = [P]
3. Methyl orange end point = [M]

[P] = NaOH + ½ Na_2CO_3

[M] = NaOH + Na_2CO_3

Na_2CO_3 = 2{[M] – [P]} = V_1

NaOH = M – 2{[M] – [P]} = V_2

GENERAL CALCULATIONS

Standadisation of HCl

Volume of alkali solution (Na_2CO_3) taken = 10 ml

Normality of $Na_2CO_3 = N_2$

Volume of HCl used = V_o

Using the normality equation

$N_1 \times V_o = N_2 \times 10$

$N_1 = N_2 \times (10/V_o)$

Determination of Na_2CO_3 and NaOH

(i) Determination of NaOH

Equivalent weight of NaOH = 40

Hence 1L of 1N HCl = 40g of NaOH

Normality of HCl used = N_1

V_2 ml of N_1 HCl = $40 \times (V_2/1000) \times N_1$

= y_1 gm of NaOH.

This is the amount of NaOH present in 10mL of the give alkali mixture solution.

Strength of NaOH = $y_1 \times 1000/10 = 100 \times y_1$ gm/L.

(ii) Determination of Na_2CO_3

Equivalent weight of $Na_2CO_3 = 53$

Hence 1L of 1N HCl = 53 gm of Na_2CO_3

Normality of HCl used = N_1

V_1 mL of N_1 HCl = $53 \times (V_1/1000) \times N_1$

= y_2 gm of Na_2CO_3.

This is the amount of Na_2CO_3 present in 10mL of the given alkali mixture solution.

Strength of $Na_2CO_3 = y_2 \times (1000/10) = 100 \times y_2$ gm/L.

RESULTS: The given alkali mixture contains NaOH = $100 \times y_1$ gm/L

The given alkali mixture contains $Na_2CO_3 = 100 \times y_2$ gm/L.

Expected CLOs/Daily life application: The total basic content of an antacid tablet can be determined in a similar manner.

FORMAT (for recording observations/ burette readings) [to be written on the left hand side of practical note book]

OBSERVATIONS

(i) Standardization of HCl solution

Volume of 0.1 N Na_2CO_3 solution taken for each titration = 10 mL

Sr. No.	Burette reading (mL)		Volume of HCl used (mL)
	Initial	Final	
1.			
2.			
3			
4			

Mean volume of HCl used = _____ (mL)

(ii) Determination of NaOH and Na_2CO_3 in the mixture

Volume of mixture of NaOH and Na_2CO_3 solution taken for each titration = 10 mL

Sr. No.	Burette Reading (mL)			Volume of HCl used (mL)	
	Initial (A)	Colourless with Phenolphthalein (B)	Reddish colour with Methyl orange (C)	[P] = B – A	[M] = C – A
1					
2					
3					
4					

Mean volume of HCl used for [P] = _____ (mL)

Mean volume of HCl used for [M] = _____ (mL)

EXPERIMENT NO. 5

EXPERIMENT: To determine the molecular weight of a polymer (polystyrene) by using Ostwald's viscometer.

APPARATUS: Ostwald's viscometer, stop watch.

CHEMICALS: Polystyrene, toluene.

THEORY: When a very small amount of a polymer is added to a solvent of low viscosity, the viscosity of the resulting solution increases sharply. This increase in viscosity depends upon the molecular weight of the polymer, concentration, size and shape of the solute molecules.

*The relative viscosity (η_r) of the polymer solution will be given by

$$\eta r = \frac{\eta_s}{\eta_0} \qquad \text{……(i)}$$

where η_s is the coefficient of viscosity of the polymer solution.

η_0 is the coefficient of viscosity of the pure solvent at the same temperature.

Now $$\frac{\eta s}{\eta 0} = \frac{P_s\ t_s}{P_0 t_0} \qquad \text{.....(ii)}$$

where P_s and P_0 are the densities of polymer solution and the pure solvent, and ts and t0 are the corresponding time of flow of specified volume through the capillary in viscometer.

For a dilute solution, it can be assumed that $P_s = P_{0'}$ hence equation (ii) becomes

$$\eta r = \frac{t_s}{t_0} \qquad \text{…… (iii)}$$

Further, the specific viscosity (η_{sp})of the polymer solution is obtained by:

$$\eta_{sp} = \frac{\eta s - \eta_0}{\eta_{sp}} \qquad \text{……(iv)}$$

where $\eta s - \eta_0$ is the increase in viscosity of the solvent due to the presence of a solute.

or $$\eta_{sp} = \frac{\eta_s}{\eta_0} - 1 \qquad \text{…….(v)}$$

$$= \eta_r - 1 = \frac{t_s}{t_0} - 1 \qquad \text{……(vi)}$$

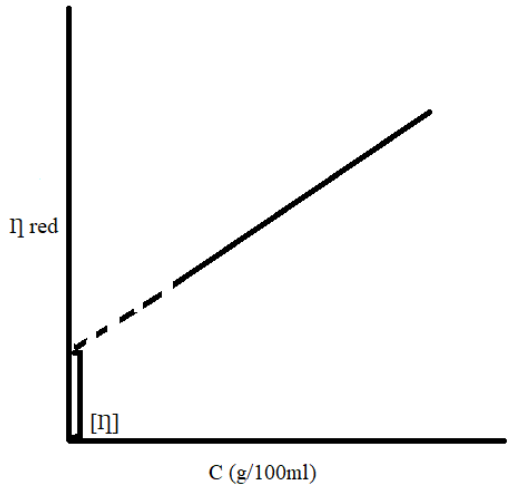
The ratio of specific viscosity of the solution of its concentration C (expressed in g/100ml) is known as *Viscosity Number* or *Reduced Viscosity* η_{red}.

$$\eta_{red} = \frac{\eta_{sp}}{C} \qquad \text{…….(vii)}$$

The plot of η_{red} versus concentration is a straight line. It is given by the equation,

$$\eta_{red} = \frac{\eta_{sp}}{C} = mC + \text{constant} \qquad \text{.....(viii)}$$

where m represents the slope of the line.



Plot of η_{red} versus concentration.

The value of constant in equation (viii) is given by the intercept on the coordinate, which is obtained by extrapolating the graph to zero concentration or infinite dilution. This is termed as *limiting Viscosity* or *Intrinsic Viscosity* [η]. Actually, intrinsic viscosity is the limiting value of reduced viscosity

Thus, $[\eta] = \lim_{C\to 0}\left(\frac{\eta_{sp}}{C}\right) = \lim_{C\to 0}\left(\frac{\eta_s - \eta_0}{\eta \times C}\right)$ .....(ix)

For linear polymers, the intrinsic viscosity [η] is related to the molecular weight of the polymer by *Mark-Kuhn-Houwink equation*:

$$[\eta] = KM^a \qquad \text{.....(x)}$$

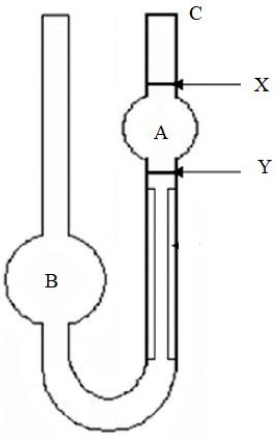
Where Mis the average molecular weight of the polymer; K and a are constants for a given polymer-solvent system.

Thus, molecular weight of the polymer can be determined from viscometric measurements using equation (x). For most systems 'a' lies between 0.6 to 0.8.

PROCEDURE:

1. Weigh accurately 0.5g of polystyrene and transfer it in a 100ml measuring flask.
2. Add about 90-95 ml toluene and dissolve polystyrene by shaking it. Make up the volume to 100ml to prepare 0.5% of the solution.

3. Similarly prepare the solutions of different concentrations (0.1%, 0.2%, 0.3% and 0.4%) by diluting the above solution.
4. Wash the Oswald's viscometer with chromic acid, distilled water and dry in an oven. Pipette out 20 ml of the pure solvent (toluene) in the bulb B of the viscometer which is clamped vertically in a stand, placed in thermostat at 25^o C or 30^o C.



5. Through a rubber tube attached to upper arm of bulb A, suck up the solvent until it rises above the mark X.
6. Allow the solvent to fall freely through a capillary upto the mark X. Start the stop watch and note the time t_1 for the flow of liquid from mark X to mark Y.
7. Repeat to get five readings and take the mean as the flow time t_0.
8. Remove the solvent, clean the viscometer and dry it.
9. Pipette out 20 ml of one of the solutions prepared above (say 0.5%) and determine the flow time as above. Repeat to get three readings and take their mean.
10. Similarly, determine the flow times for solutions of different concentrations after proper cleaning and drying of the viscometer after each set of readings.

OBSERVATIONS:

Temperature of the experiment =

Solvent used =

Value of constant 'K' for styrene/ solvent at the above temperature (from the table) =

Value pf constant 'a' (from the table) =

S. No.	Concentration of polymer solution	Flow time (Mean of three readings)	Relative viscosity $\eta_r = \eta_s/\eta_0 = t_s/t_0$	Specific viscosity $\eta_{sp} = \eta_r - 1$	Reduced viscosity $\eta_{red} = \eta_{sp}/C$
1.	Pure solvent	t_0			
2.					
3.					
4.					

GENERAL CALCULATIONS:

Plot the graph between η_{red} and concentration and extrapolate the graph to zero concentration.

Find out the value of intrinsic velocity [η] from the graph.

$$[\eta] = \text{Intercept}$$

Now, we know, $[\eta] = KM^a$

substitute the values of [η], K, and a and calculate M, the molecular weight of the polymer.

Using logarithm,

$$\log [\eta] = \log K + a \log M$$

$$\log M = \frac{\log [\eta] - \log K}{a}$$

$$M = \text{Antilog} \frac{[\log [\eta] - \log K]}{a}$$

RESULT: The molecular weight of the given polymer (polystyrene) is …….

PRECAUTIONS:

1. All measurements should be made using a constant volume of the liquid.
2. Viscometer should be kept perfectly vertical.
3. Mouth suction should not be done.
4. There should not be any air bubbles in the viscometer.

Table Values of ‘K’ and ‘a’

Polymer	**Solvent**	**Temperature**	**‘K × 10^4’**	**‘a’**
Polystyrene	Toluene	25	1.1	0.72
		30	1.1	0.725

EXPERIMENT NO. 6

EXPERIMENT: To determine the copper content of a given sample of copper ore solution using 0.1 N sodium thiosulphate iodometrically.

APPARATUS: Pipette, burette, beakers, conical flask, funnel, burette stand and clamp.

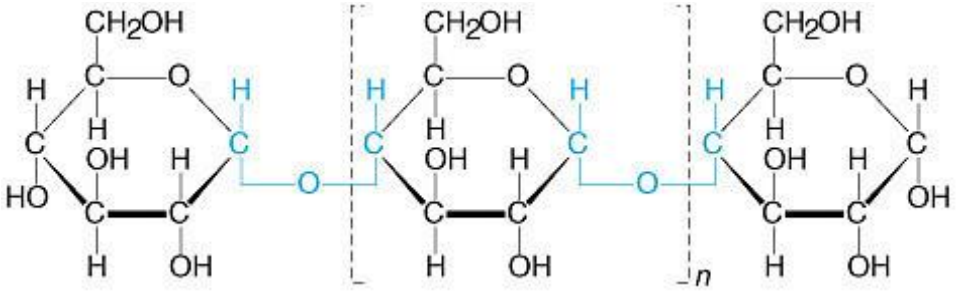
CHEMICALS: Copper sulfate ($CuSO_4$), solid sodium bicarbonate ($NaHCO_3$), acetic acid (CH_3COOH), potassium iodide (KI), starch solution and sodium thiosulfate ($Na_2S_2O_3$).

THEORY: Estimation of copper in the copper ore is based on the fact that copper can quantitatively liberate iodine from potassium iodide solution in an acidic medium. The liberated iodine can be titrated against a given standard sodium thiosulphate solution using starch as an indicator.

$$2\,CuSO_4 + 4KI \xrightarrow{H^+} 2\,CuI_2 + 2\,K_2SO_4$$

$$2CuI_2 \longrightarrow Cu_2I_2\downarrow + I_2$$

$$2\,Na_2S_2O_3 + I_2 \longrightarrow 2\,Na_2S_4O_6 + 2NaI$$



Starch

End point is the appearance of white color due to precipitates of Cu_2I_2. As Cu_2I_2 is soluble in mineral acids but insoluble in weak organic acids (acetic acid), the strongly acidic medium is neutralized with $NaHCO_3$ till a faint permanent precipitates of basic copper carbonate are formed which are dissolved with a few drops of acetic acid.

PROCEDURE

1. Pipette out 10 ml of the copper ore solution into a titration flask.

2. Add small amount of some solid $NaHCO_3$ to the ore solution in small doses till there is no effervescence. The solution turns milky at this stage.

3. Add dilute acetic acid dropwise, just sufficient to remove the milkiness. To the clear blue solution, add 5 ml of 10 % KI solution. Color of the solution changes to ***dark brown***, due to the formation of KI_3.

4. Add about 35 ml of distilled water to dilute the contents of the flask. Wait for atleast 3 minutes. Titrate the solution against standard sodium thiosulphate solution till the ***color turns to pale/ light yellow.***

5. Add about 2 ml of 1% freshly prepared starch solution. Color of the solution turns to ***deep blue.***

6. Continue the titration (**same conical flask**) with more sodium thiosulphate solution till the ***color changes from blue to permanent white***.

7. Keep the contents of the flask for some time on the table-shelf. It should not turn blue again. If this happens, add a few more drops of $Na_2S_2O_3$ solution to get permanent white color again.

8. Repeat the experiment to get atleast five correct readings till atleast two concordant readings are obtained.

OBSERVATIONS

Volume of copper ore solution taken for each titration = 10 ml

Sr. No.	Burette Reading (mL)		Volume of 0.1 N $Na_2S_2O_3$ soln. added (mL)
	Initial	Final	
1.			
2.			

Mean volume of $Na_2S_2O_3$ used = (V_2) _____ mL

GENERAL CALCULATIONS

Volume of copper ore solution used for each titration (V_1) = 10ml

Normality of sodium thiosulphate solution = 0.1 N

Let volume of $Na_2S_2O_3$ used = V_2 ml

Applying the normality equation

Copper ore $Na_2S_2O_3$

$N_1V_1 = N_2V_2$

10 ml of N_1 copper ore solution = V_2 ml of 0.1 N $Na_2S_2O_3$ solution

N_1 [Normality of copper solution] = $(0.1 \times V_2)/10 = V_2$

Eq. wt. of copper = 63.50

Amount of copper in the given ore = $N_1 \times 63.5$ gm/L

RESULT: The amount of the copper present in copper ore solution is ____ gm/L.

PRECATIONS

1. The white color at the end point should be permanent.
2. The copper ore solution should be neutralized before titration.
3. The contents of the titration flask should be diluted to observe better change of color at the end point.
4. After mixing the initial solutions, wait for atleast 3 minutes before starting the titration.
5. General precautions of volumetric titrations should be followed.

Expected CLOs/Daily life application: As both excess and low levels of copper can have adverse effect, e.g., excess of copper in body can cause “Wilson Disease”. Hence copper levels should be checked regularly.

EXPERIMENT NO. 7

EXPERIMENT: Determine the strength of sodium hydroxide solution by titration with standard hydrochloric acid (0.1 N) conductometrically.

APPARATUS: Pipette, burette, beakers, funnel, burette stand, clamp, conductometer and conductivity cell.

CHEMICALS: Standard hydrochloric acid (HCl) and sodium hydroxide (NaOH).

THEORY: There is a replacement of the H^+ ion with an equivalent amount of Na^+ upon the addition of NaOH solution to the HCl solution, resulting in a decrease in the conductivity of the solution since the ionic mobility of Na^+ is smaller than H^+ ions.

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

During titration, the conductivity of the solution first decreases up to the equivalence point, then increases due to an increase in hydroxyl ion concentration. Initially, with the addition of the alkali to the acid, there will be a decrease in conductance. After the neutralization is complete, further addition of alkali would result in increase of conductance, since the additional OH^- ions from NaOH are no longer used up in the chemical reaction. So, if we plot conductivity versus volume of titrant/ NaOH, we get V shaped curve. From the titration curve an equivalence point can be obtained.

PROCEDURE

1. Take 50 ml of HCl solution in a clean beaker and immerse/dip the conductivity cell in it. Make sure that the two platinum electrodes of the cell are completely dipped in the solution.
2. Connect the cell to the bridge. Note down the conductivity.
3. Add NaOH from the burette at an interval of 0.5 ml each time, stir the contents and note down the conductivity every time. The conductivity will first decrease and then increase.
4. Plot the conductance against the volume of NaOH added. The equivalence point can be determined from the inter-section of two lines on the graph and hence the strength of NaOH

solution can be calculated. This procedure can also be applied to find the strength of mixtures of two acids or bases and also in the precipitation titration.

OBSERVATIONS

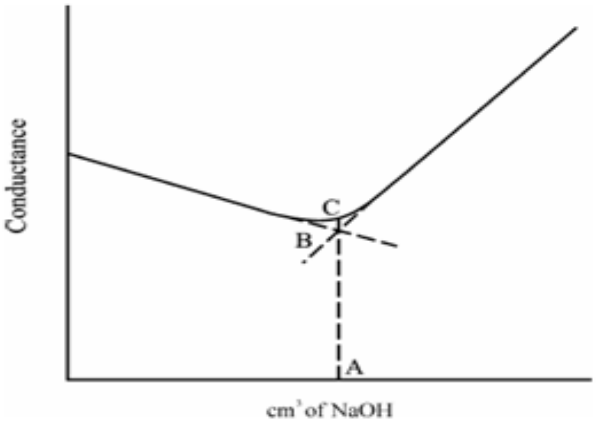
Volume of 0.1 N HCl solution taken in the beaker = 50 ml

Vol. of NaOH added from the Burette (mL)	Conductivity (millimho)

Applying Normality Equation:

$$N_{NaOH} = N_{HCl} \times \underline{(50/V_{NaOH})}$$

Strength of NaOH (gm/L) = Normality × equivalent weight



A= Equivalence Point

RESULT: The strength of sodium hydroxide present in the given sample is _____ gm/L

Expected CLOs/Daily life application:

- It can be used to check water pollution in lakes as well as rivers.
- It is also used to check the alkalinity of the fresh water.
- Salinity of the sea water can also be checked by this method.
- Used for tracing microorganism in food microbiology.
- To check the solubility of sparingly soluble salts.

EXPERIMENT NO. 8

EXPERIMENT: Determine pKa value of acetic acid by pH metric titration

APPARATUS: Pipette, burette, beakers, funnel, burette stand, clamp, pH meter and glass electrode.

CHEMICALS: Sodium hydroxide (NaOH) and acetic acid (CH_3COOH).

THEORY: A pH meter will be used to follow the titration of an unknown weak acid, HA (aq) with sodium hydroxide. NaOH (aq).

$$HA\ (aq) + NaOH\ (aq) \longrightarrow NaA\ (aq) + H_2O$$

The weak acid has a concentration around 0.1M. The result of the pH versus volume of NaOH plot is "S" shaped curve which is not as steep as the one arising from the titration of a strong acid. The equivalence point (this time) will be at alkaline pH (not 7 as in strong acid vs strong base). From the equivalence point, the concentration of an unknown acid HA is found. In addition, the acid constant K_a can be determined.

$$HA + H_2O \longrightarrow H_3O + A^-$$

$$pH = pKa + \log \frac{[\text{salt form}]}{[\text{acid form}]}$$

Henderson–Hasselbalch equation

$$K_a = \frac{[H_3O^+]\,[A^-]}{[HA]}$$

PROCEDURE

Titration of unknown HA with standard NaOH

1. Calibrate the pH meter with the standard buffer solution of pH = 4 or 9, then rinse the glass electrode and immerse it in the beaker. Position the burette so that the titrant can be easily added.
2. Pipette out 50 ml of acetic acid into a clean beaker, dip the glass electrode. Record the pH.
3. Initially, add 0.5 ml of 0.1 N NaOH solution at a time, record the pH (after each addition), until the pH change is more than 0.2–0.3 units, then start adding 0.2 ml of NaOH each time (i.e., near to the equivalence point, decrease the volume of NaOH added) so that the change in pH is small enough to yield a good shape of plot.
3. After the rapid change in pH (after the equivalent point), the volume of NaOH may again be increased to 0.5 ml per addition. Make at least 10 more additions after the equivalence point so that the region with the plateau can be plotted.

4. pK_a is determined by examining the titration curve. The negative log of K_a is pKa and is same ***as the pH at half the volume of equivalence point.***

(pH = pKa when logarithm term is zero which in turn is zero once [salt] = [acid]. This is true at half equivalence point) cf. Henderson–Hasselbalch equation.

OBSERVATIONS

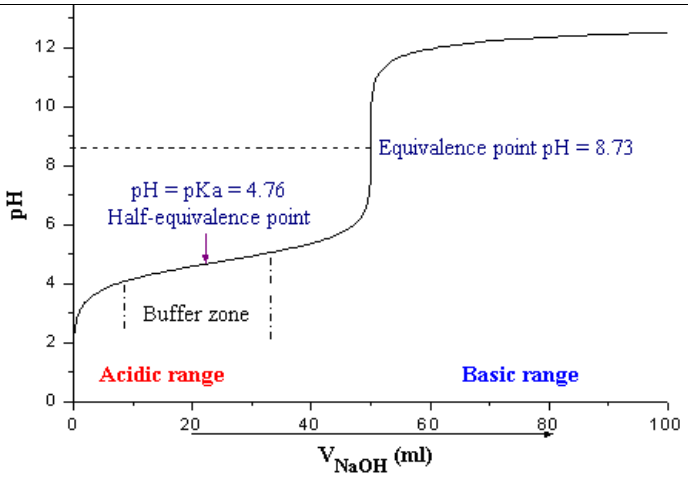
Normality of standard NaOH = 0.1 N

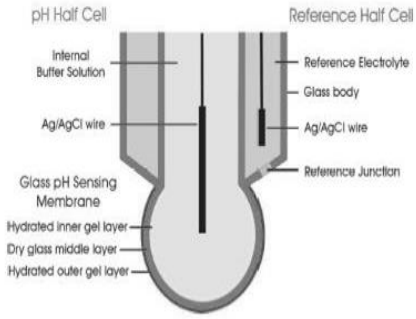
Vol. of NaOH added from the Burette (mL)	pH

Using graph, the volume of NaOH at equivalence point and half equivalence point can be determined, as shown in the sample graph below.

Sr. No	NaOH (mL) at the equivalence point	NaOH (mL) at the half equivalence point	pH (at the half equivalence point) = pKa

Sample graph and Line diagram of glass electrode:





Line diagram of Glass electrode

RESULT: The pKa of acetic acid is _____.

Expected CLOs/Daily life application: Knowledge of pK_a values is important for the quantitative treatment of systems involving acid–base equilibria in solution. Design of buffers (that resist any change in pH), drug development are couple of applications that require knowledge of pK_a.

EXPERIMENT NO. 9

EXPERIMENT: To titrate potentiometrically ferrous ammonium sulphate solution against potassium permanganate and to determine the standard electrode potential of ferrous-ferric system.

APPARATUS: Pipette, burette, beaker, funnel, burette stand, clamp, potentiometer, calomel electrode (or Ag/AgCl electrode) and platinum electrode.

CHEMICALS: Mohr's salt solution (Ferrous ammonium sulfate; $FeSO_4(NH_4)_2SO_4.6H_2O$), potassium permanganate ($KMnO_4$) and sulphuric acid (H_2SO_4).

THEORY: An electrochemical cell is a device which establishes measurable electrical potential differences and in which flow of electrical current is accompanied by an overall chemical change. A reversible cell is that in which the overall chemical reaction can be reversed in the presence of an opposing external electromotive force of magnitude greater than that if cell itself. An electromotive cell consists of two electrodes or half cells, whose electrolytic solutions are either directly in contact with each other or connected through an intervening electrolytic solution. The net chemical change takes place at the individual electrodes; one of which is oxidation and the other reduction.

At the reversible electrodes, the oxidized and reduced states of a system exist in equilibrium in the solution, where an inert metal electrode (like Pt) is dipped into it e.g., Fe^{3+}/Fe^{2+}, in which the reaction is:

$$Fe^{3+} + e^- \longrightarrow Fe^{2+}$$

The experimental cell:

$$Hg, Hg_2Cl_2(s)/KCl(saturated) \parallel Fe^{3+}/Fe^{2+}/Pt \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ (1)$$

The e.m.f. of the cell:

$$E = E^0_{(Fe^{3+}/Fe^{2+})} + \frac{2.303\,RT}{nF}\log\frac{[Fe^{3+}]}{[Fe^{2+}]} - E_{(calomel)} \qquad \ldots\ldots\ldots\ldots\ldots\ (2)$$

Chemical reaction during potentiometric titration:

$$5\,Fe^{2+}_{(aq)} + MnO_4^-{}_{(aq)} + 8\,H^+ \longrightarrow 5\,Fe^{3+}_{(aq)} + Mn^{2+}_{(aq)} + 4\,H_2O_{(l)}$$

When potassium permanganate solution is added to Mohr's salt solution, the concentration of Fe^{2+} ions decreases and that of Fe^{3+} ions increases, and as a result the emf of the cell increases slowly. Near to the equivalence point, an inflection in seen due to fall in concentration of Fe^{2+} ions ultimately to 0, resulting in sudden rise in emf of the cell.

At half equivalence, eq. 2 becomes

$$E_{(\text{half equivalence})} = E^o_{(Fe^{3+}/Fe^{2+})} - E_{(\text{calomel})}$$
$$= E^o_{(Fe^{3+}/Fe^{2+})} - 0.242 \text{ (at 298 K)} \quad \ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Thus, by noting $E_{(\text{half equivalence})}$ from the graph, $E^o_{(Fe^{3+}/Fe^{2+})}$ can be calculated using eq. 3.

PROCEDURE

1. Take 50 ml of 0.1 N FAS solution in the beaker and add 5 ml of 1N sulphuric acid. Dip the platinum and saturated calomel electrodes in the solution.
2. Connect the indicator and the reference electrodes to the black and red terminals of the potentiometer, respectively.
3. Rinse and fill the burette with $KMnO_4$ (0.2 N).
4. Note the initial emf of the cell and start addition of the titrant ($KMnO_4$) in portions of 1 mL each. Near the equivalence point, decrease the volume of additional titrant to 0.5 ml and later on to 0.2 ml and note down to reading after each addition.
5. Continue to take 10-12 readings more, after a sharp change/increase in emf is noticed.
6. Plot emf (in volts) against the volume of $KMnO_4$ solution added (mL) and note down the equivalence point and the potential at half the equivalence point.

OBSERVATIONS

(i) Vol. of $KMnO_4$ Vs. e.m.f. of the solution

Vol. of $KMnO_4$ added from burette (mL)	e.m.f. (V)

S. No.	Vol. of $KMnO_4$ (mL) at equivalence point	Vol. of $KMnO_4$ (mL) at half equivalence	e.m.f. (V) at half equivalence
1.	X (along x-axis)	X/2 (along x-axis)	**Y** (along y-axis)

GENERAL CALCULATIONS

The e.m.f. at half-equivalence point (**Y**) is observed from graph. The graph is very similar in shape (S-shape) to the graph obtained in experiment 8.

$E_{(\text{half equivalence})} = E^{o}_{(Fe^{3+}/Fe^{2+})} - E_{(\text{calomel})}$

$E^{o}_{(Fe^{3+}/Fe^{2+})} = E_{(\text{half equivalence})} + E_{(\text{calomel})}$

$E^{o}_{(Fe^{3+}/Fe^{2+})} = \mathbf{Y} + 0.242 =$ _____ V

RESULT: The standard half-cell potential of Fe^{3+}/Fe^{2+} couple is _____ V.

PRECATIONS

1. After each addition of the titrant/ $KMnO_4$, the contents of the beaker should be stirred gently.
2. Electrodes should be handled very carefully.

Expected CLOs/Daily life application: To quantify alkalinity, acid content, and chloride ion, fluoride ion and various other ions in water, fertilizers, soil etc. Solubility product (K_{sp}) of salts can also be determined using potentiometry.

EXPERIMENT NO. 10

EXPERIMENT: Spectrophotometric determination of iron(II) with 1,10-phenanthroline.

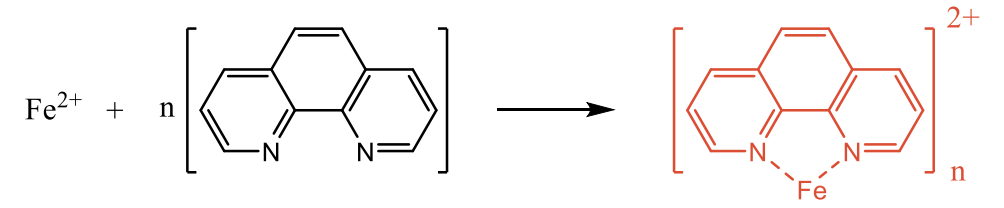
APPARATUS: Burette, volumetric flasks (50 mL), cuvettes, funnel, burette stand, clamp and colorimeter

CHEMICALS: Mohr's salt solution (Ferrous ammonium sulfate; $FeSO_4(NH_4)_2SO_4.6H_2O$), 1,10-phenanthroline, hydroxylamine hydrochloride, acetic acid-sodium acetate buffer of pH 4.5 and sulphuric acid (H_2SO_4).

THEORY: Iron(II) reacts with 1,10-phenanthroline to form an ***orange red*** complex $[(C_{12}H_8N_2)_3Fe]^{+2}$. The color intensity is independent of the acidity in pH range 2-9. If iron (III) is present it can be reduced with hydroxylamine hydrochloride. The absorbance is mentioned at a wavelength of 515 nm. The variation in the concentration of a given colored solution, changes the intensity of the transmitted light. The change in light intensity is measured by the instrument called photocolourimeter/ colourimeter. When monochromatic light falls on a solution sample, some light is absorbed and the intensity of the transmitted light is decreased. The decrease in intensity of light is proportional to the thickness of the absorbing medium and the concentration of solution. This may be expressed by Lambert-Beer law:

$$A = \log(1/T) = \log(I_o/I) = \varepsilon.b.c$$

Where c is the concentration of the solution expressed in mol/L and 'ε' is a constant characteristic of the solute and the wavelength of light, ε is called the molar extinction coefficient. 'A' is absorbance or optical density (D) of solution; *b* is path length and is related to the transmittance ($T = I/I_o$).



Colourless — Phenanthroline (Colourless) — Iron-phenanthroline complex (Reddish-orange colour)

n = number of phenanthroline molecules reacting with Fe^{2+}

PROCEDURE

A. Preparation of Samples

1. Take six 50 mL volumetric flasks and add 0, 1, 2, 3, 4, 5 ml of FAS solution in each flask. Let's name these volumetric flasks as **K, L, M, N, O and P.**
2. Then add 2 ml of 1,10-phenanthroline solution to each of these volumetric flasks.
3. Now dilute each volumetric flask with deionised water to afford a total volume of 50 mL (by filling the these flasks upto the mark). Stopper the flasks and mix the contents well by shaking vigorously for few minutes. Allow the solution to stand for 10 minutes.
4. The first volumetric flask to which 0 ml of FAS is added (i.e., no Fe^{2+} ions), will serve as a blank. (**Solution K**).

The Fe concentration in these flasks will be:

K	0.0 N	**N**	6.0×10^{-5} N
L	2.0×10^{-5} N	**O**	8.0×10^{-5} N
M	4.0×10^{-5} N	**P**	10.0×10^{-5} N

B. To determine the λ_{max}

1. Get the two cuvettes issued from the laboratory staff.
2. Fill one of them with the blank solution (**K**) and another one with the one of the samples containing Fe. Let's say **solution P.**
3. Light of single wavelength can be produced by selecting the filter on the photocolorimeter. Usually, the range goes from 410 nm–700 nm.
4. Set the filter to 410 nm. Place the cuvette filled with blank solution, **K**, in the sample holder.
5. Set the absorbance to 0%.
6. Now place the second cuvette, with solution **P**, in the sample holder. Measure the absorbance of the solution. Now you have the absorbance at 410 nm for solution **P**.
7. **By changing the filter to next wavelength each time, repeat steps 4–6. You need to set the absorbance to zero with blank (K) every time you change the wavelength with filter.**
8. Now, you have absorbance of solution P, over a range of wavelength from 410 nm-700 nm. You will notice that graph between the Absorbance and wavelength takes an ***inverse parabola shape***, with a maximum absorbance around 500 nm or 480 nm. This is your λ_{max}**.**

C. Measurement of Absorbance for Solutions L to P at λ_{max}

1. Set the filter to λ_{max} obtained in part B (step 8).
2. Set the absorbance to 0 using your blank sample (**K**).
3. Measure the absorbance for solutions **L** to **P** now at λ_{max}. Don't disturb the filter in between.
4. **Now measure the absorbance for an unknown sample provided to you.**
5. Plot absorbance vs. concentration for samples **L** to **P**. Connecting maximum points, draw a straight line ideally passing through origin.
6. Using absorbance value for the unknown, find out its concentration.

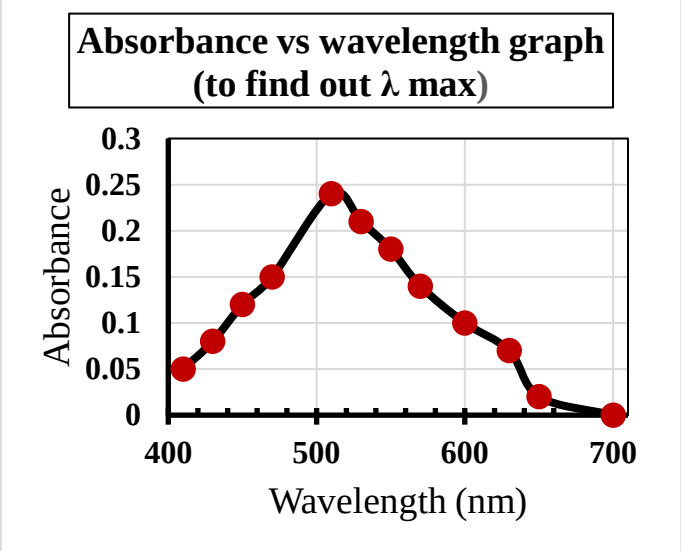
OBSERVATIONS

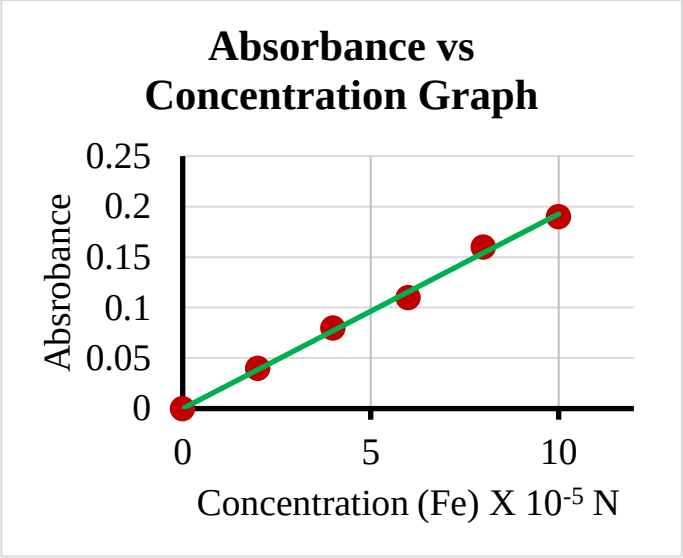
(i) Absorbance of the solution at highest concentration (10 ×10^{-5} N) at various λ

Wavelength (nm)	Absorbance

(ii) Absorbance of the solutions at different concentration at λmax

Concentration (N)	Absorbance





Sample UV-Vis and calibration curve (for the determination of concentration of unknown)

The concentration of unknown can be determined from its absorbance value using second graph. A solution with Fe concentration of 2.00×10^{-5} N contains 10.00 μg of Fe (as used in this experiment). Hence knowing the Fe concentration of unknown samples, their Fe content in μg can be calculated.

RESULT: The Fe content in the unknown / given sample is _____ μg of Fe

Expected CLOs/Daily life application: Using colorimeter, the quantification of various metal ions, ligands can be done. It can also be used to extract the value of binding constant. Kinetics of a reaction can be measured in the colorimeter.

EXPERIMENT NO. 11

EXPERIMENT: Determination of cloud and pour point of given oil sample

APPARATUS: Cloud and pour point apparatus, thermometer and ice cubes.

CHEMICALS: Coconut oil, Petroleum

THEORY: 'Cloud point' and 'Pour point' are the two parameters which determine the quality of oil. Cloud point parameter is limited to only oils which are transparent. Lubricating oils obtained from petroleum usually contains paraffin wax and other asphaltics impurities, their amount depending upon the efficiency of refining and de-waxing processes. When petroleum is chilled under specific conditions, the temperature at which paraffin wax or other solidifiable materials (normally dissolved in oil) begin to separate out from solution in the form of minute crystals, causing the oil to become less transparent, cloudy or hazy in appearance is known as the cloud point of the oil. If the cooling is continued further, the amount of separating oil increases and a stage is reached at which the oil solidifies and stops flowing. The lowest temperature at which the oil will not flow or pour under the prescribed conditions, when cooled undisturbed at a fixed rate is called its pour point.

PROCEDURE

(A) Determination of Cloud Point

(i) Bring the oil sample to be tested to a temperature at least 15 ^{o}C above the expected cloud point. If the sample contains moisture, dry it by shaking with a little anhydrous sodium sulphate followed by filtration.

(ii) Pour the clear oil into test jar upto etched mark.

(iii) Tightly close the jar with a cork carrying thermometer with a bulb touching the bottom of the jar.

(iv) Insert the test jar inside a holding jacket (made of glass or copper), which is immersed in freezing mixture suitable for obtaining the desired temperature.

(v) After every 2 ^{o}C fall in temperature of oil, take the oil sample out of the test jar from the jacket. Inspect the cloudiness and immediately replace it in the jacket.

(vi) Record the temperature of such inspection at which it first reveals a distinct cloudiness in oil sample near to the bottom of jar and report it as the cloud point.

(B) Determination of Pour Point

(i) The same procedure as above is followed for cooling the oil except that the thermometer bulb is just completely immersed in the oil.

(ii) Take out the test jar after every 3 ^{o}C fall in temperature and tilt it just enough to see any movement of oil. Immediately replace the jar in jacket.

(iii) A point (temperature) at which oil does not show any movement in jar on tilting and holding the jar in horizontal position for 5 seconds.

(iv) Record the reading on the test thermometer as solid point.

(v) Add 3 ^{o}C to this temperature and report that as pour point.

OBSERVATIONS

Cloud point = _____ ^{o}C

Solid point = _____ ^{o}C

Pour point = Solid point + 3 ^{o}C

RESULTS

Cloud Point = _____ ^{o}C

Pour point = Solid point + 3 ^{o}C

PRECATIONS

(1) The test jar should not touch the jacket. This is achieved by placing a cork disk at the bottom of jacket and using a gasket around the test jar.

(2) The complete operation, removal and replacement of test jar should not take more than 3 seconds.

(3) After the cloud point reaches, the mass of oil should not be disturbed as this may result in delay solidification and so lower value of the result will be obtained.

Expected CLOs/Daily life application: Cloud point helps to determine the minimum operating temperature of an automobile etc.

Hope you enjoyed the Laboratory Experiments of Chemistry Course (UCB009)

All The Best!